INTERNATIONAL STANDARD

ISO 439

Third edition
2020-02

Steel and cast iron — Determination of silicon content — Gravimetric method

Aciers et fontes — Détermination des teneurs en silicium — Méthode gravimétrique

Reference number
ISO 439:2020(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This third edition cancels and replaces the second edition (ISO 439:1994), which has been technically revised. The main change compared to the previous edition is:

— a complete reevaluation of the precision data.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steel and cast iron — Determination of silicon content — Gravimetric method

1 Scope

This document specifies a gravimetric method for the determination of the silicon content in steel and cast iron.

The method is applicable to silicon contents between 0,10 % (mass fraction) and 5,0 % {mass fraction).

NOTE For samples containing molybdenum, niobium, tantalum, titanium, tungsten, zirconium or high levels of chromium, the results are less accurate than for unalloyed matrixes.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Dissolution of a test portion with hydrochloric and nitric acids.

Conversion of acid-soluble silicon compounds to hydrated silicon dioxide by evaporation with perchloric acid until white fumes appear. Filtration of the hydrated silicon dioxide and acid-insoluble silicon compounds, ignition to form impure silicon dioxide and then weighing.

Treatment of the ignited residue with hydrofluoric and sulfuric acids, followed by ignition and weighing.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and grade 2 water as specified in ISO 3696.

5.1 Hydrochloric acid, ρ approximately 1,19 g/ml.

5.2 Hydrochloric acid solution, 1 + 1.

Add 500 ml of hydrochloric acid (5.1) to 500 ml of water and mix.

5.3 Hydrochloric acid solution, 1 + 19.

Add 10 ml of hydrochloric acid (5.1) to 190 ml of water and mix.

5.4 Nitric acid solution, 3 + 1.

Add 150 ml of nitric acid, ρ approximately 1,40 g/ml, to 50 ml of water and mix.

5.5 Hydrofluoric acid, ρ approximately 1,14 g/ml.

5.6 Perchloric acid, ρ approximately 1,67 g/ml.

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

NOTE Perchloric acid (ρ approximately 1,54 g/ml) can also be used.

5.7 Sulphuric acid solution, 1 + 1.

Cautiously add 50 ml of sulphuric acid, ρ approximately 1,84 g/ml, to 50 ml of water, allow to cool and mix.

6 Apparatus

Ordinary laboratory equipment and the following shall be used:

6.1 Platinum crucibles, of capacity approximately 30 ml.

6.2 Muffle furnace, adjustable from 800 °C up to 1 100 °C.

6.3 Filter paper, medium-texture, of known low ash content.

7 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steels and cast irons.

8 Procedure

8.1 Test portion

Use millings or drillings of a maximum thickness of 0,2 mm.

According to the presumed silicon content, weigh, to the nearest 1 mg, the following mass (m_0) of the test portion:

a) for silicon contents between 0,10 % (mass fraction) and 0,50 % (mass fraction): m_0 approximately 5 g;

b) for silicon contents between 0,50 % (mass fraction) and 2,5 % (mass fraction): m_0 approximately 2,5 g;

c) for silicon contents between 2,5 % (mass fraction) and 5,0 % (mass fraction): m_0 approximately 1 g.